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IS 1448-94 (1980): Methods of test for petroleum and its products, Part 94: Test for oxidation stability of lubricating greases by oxygen bomb method [PCD 1: Methods of Measurement and Test for Petroleum, Petroleum Products and Lubricants]

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Indian Standard

METHODS OF TEST FOR PETROLEUM AND ITS PRODUCTS

[P : 94]

TEST FOR OXIDATION STABILITY OF LUBRICATING GREASES BY THE OXYGEN BOMB METHOD

UDC 621·892 : 543·876

Adapted, with permission, from the Joint Publication No. ASTM D 942-78 and IP Designation 142/74

1. SCOPE

1.1 This standard describes the method of test for determining the resistance of lubricating greases to oxidation when stored under static conditions for long periods of time, as for example, thin coatings on antifriction bearings and on motor parts. It is not intended for the prediction of the stability of greases under dynamic service conditions nor is it intended for the prediction of the stability of greases stored in commercial containers.

2. OUTLINE OF THE METHOD

2.1 The sample of grease is oxidized in a bomb heated to 99°C and filled with oxygen at 758 kPa (7·5 kgf/cm^2). Pressure is observed and recorded at stated intervals. The degree of oxidation after a given period of time is determined by the corresponding decrease in oxygen pressure.

3. APPARATUS

3.1 Oxidation Bomb — of the type and conforming to the dimensions shown in Fig. 1, shall be made of 18 percent chromium, 8 percent nickel

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alloy steel. The bomb shall be capable of withstanding with safety a working pressure of 1241 kPa (12·4 kgf/cm^2) at 99°C and provided with a lead or TFE-fluorocarbon gasket for sealing. To facilitate cleaning, give a high polish to the interior surface of the bomb, the lid, and the inside of the pipe carrying the pressure gauge. The bomb shall be so constructed that its volume without the dish holder and dishes is 185 ± 6 ml, measured to the level of the gauge. This may be checked by assembling the bomb, removing the gauge, and measuring the amount of liquid required to fill the bomb to the level of the gauge connection. A screw-on lid may be used instead of the bolt-on lid shown in Fig. 1.

3.2 Pressure Gauge — suitable for use with oxygen and mineral oil, shall be of the indicating type, graduated in intervals of 6·89 kPa (0·1 kgf/cm^2) per scale division, with a maximum reading of at least 827 kPA (8·2 kgf/cm^2) and capable of an accuracy of at least 3·45 kPA (0·05 kgf/cm^2) within the interval 621 to 827 kPa (6·2 to 8·2 kgf/cm^2). Attach the gauge to the stem as shown in Fig. 1. A recording pressure gauge may be used in place of the prescribed indicating pressure gauge if it satisfies the volume and accuracy requirements previously stated.

3.3 Oil Bath — capable of being controlled at 99 ± 0·5°C, with a temperature gradient less than 0·5°C in the body of the oil used, and of sufficient depth to allow submersion of the bomb to the proper depth. Circulation of the oil heating medium by a pump or stirring is recommended. Sufficient heat capacity shall be provided to return the bath to the required temperature within 60 minutes after immersion of the bomb. Provide the bath with a thermometer well such that the 96·8°C point of the thermometer is at the same level as the upper surface of the bath cover. Adjust the level of the bath so that the top of the bomb is submerged approximately 50 mm below its surface. Arrange the bath so that there are no draughts or wide fluctuations in temperature around the gauges.

NOTE — Other constant-temperature baths may be used if they are equivalent in heat capacity and thermal gradient characteristics to the oil bath described above and can be shown to maintain the bomb at the prescribed test temperature.

3.4 Dish Holder — made of 18 percent chromium, 8 percent nickel alloy steel as described for the oxidation bomb, shall conform to the dimensions prescribed in Fig. 1.

3.5 Sample Dishes — of glass, conforming to the dimensions prescribed in Fig. 1.

3.6 Thermometer* — of sufficient accuracy to measure temperature to 0·1°C.

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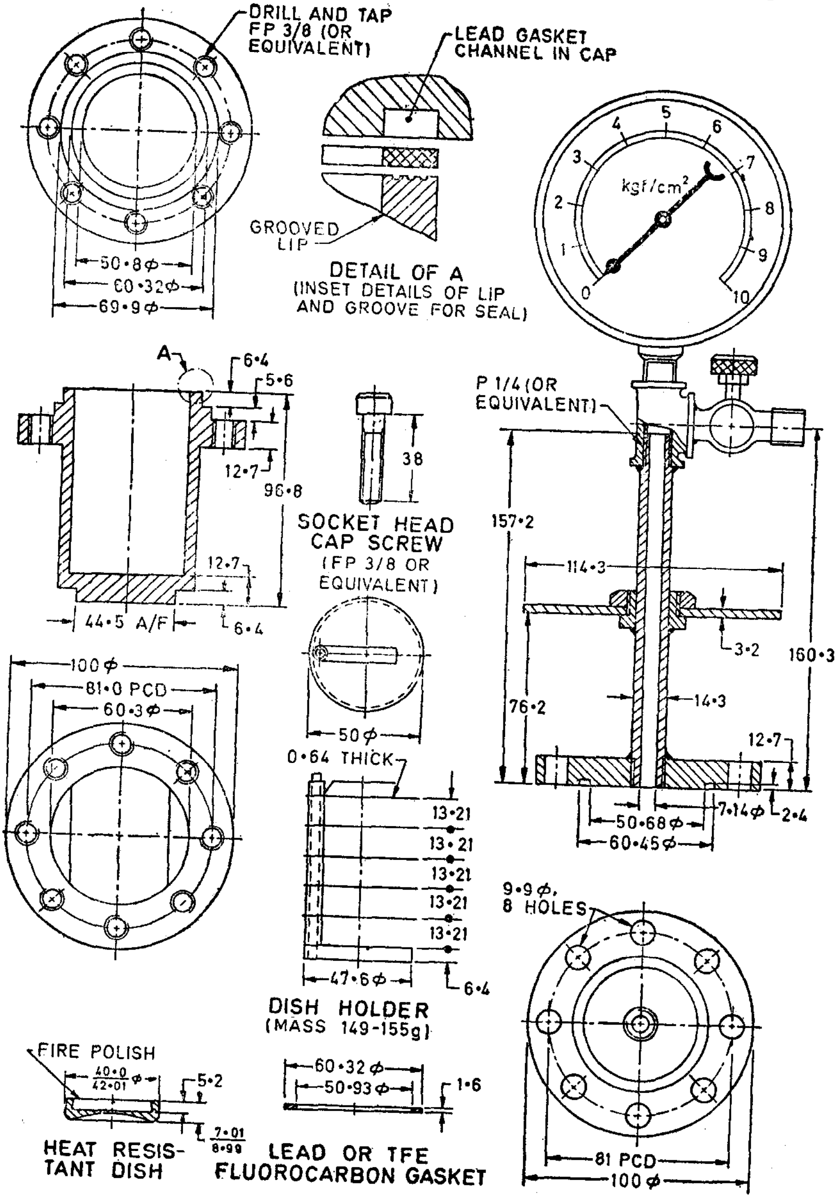
*Thermometers conforming to the specification IP 24 C may be used.



All dimensions in millimetres.

FIG. 1 APPARATUS FOR OXIDATION STABILITY TEST OF LUBRICATING GREASES

4. MATERIAL

4.1 Oxygen — of not less than 99·5 percent purity.

4.1.1 *Caution* — Since oxygen vigorously accelerates combustion, observe the following precautions:

a) Keep oxygen at high pressure away from oil and grease. Keep oil and grease away from all regulators, gauges and control equipment.

b) Use oxygen only with equipment conditioned for oxygen service by careful cleaning to remove oil and grease from area in contact with high pressure oxygen.

c) Keep combustibles away from oxygen and eliminate ignition sources.

d) Keep surfaces clean to prevent ignition or explosion, or both, upon contact with high pressure oxygen.

e) Always use a pressure regulator to deliver oxygen. Release regulator tension before opening oxygen cylinder.

f) All equipment used shall be suitable and recommended for oxygen service.

g) Never attempt to transfer oxygen from cylinder in which it is received to any other cylinder prior to use.

h) Do not drop oxygen cylinders.

j) Keep cylinder valve closed when not in use.

k) Stand away from valve when opening cylinder.

m) Do not breathe or use technical grade oxygen for inhalation purposes.

n) See other literature for details of safe practice in the use of oxygen.

5. PREPARATION OF APPARATUS

5.1 Clean the sample dishes from all contamination from previous runs and from dust settling from the air by washing them with solvent (*see* Note 1), then with hot water and soap powder, and finally with hot sulphuric acid-chromic acid solution (*see* Note 2). Follow the final cleaning operation by a thorough tap water rinse, a distilled water rinse, and drying in an oven. Handle the clean dishes only with forceps.

NOTE 1 — **Caution:** Flammable solvent. Handle in well ventilated area, preferably in a hood. Keep away from heat, sparks and open flame. Keep container closed when not in use.

NOTE 2 — **Caution:** Sulphuric acid-chromic acid. Avoid skin contact, which may cause severe burns. Wash affected areas immediately with copious amounts of water for at least 15 minutes.

5.2 If lacquer is found after a run, clean the inside of the oxidation bomb and the metal supports for the bomb dishes by immersing in hot solvent and scrubbing with a bristle brush followed by drying. Scrub further with water and a fine scouring powder until all the lacquer deposits are removed. Follow the scouring operation by a thorough tap water rinse, a distilled water rinse and drying in an oven. Handle the clean metal supports only with forceps.

6. PROCEDURE

6.1 Fill each of the five dishes with 4·00 ± 0·01 g of grease. Distribute the samples in the dishes in a uniform layer with a smooth level upper surface. Place the filled dishes on the five bottom shelves of the holder, leaving the top shelf to act as a cover to prevent condensing volatile products from dropping into the grease samples. When assembling the bomb, place a small ball of glass wool in the bottom of the stem.

6.2 Place the dish holder in the bomb and close the bomb by tightening the bolts slowly and uniformly. Clear the air from the bomb by introducing oxygen slowly until a pressure of 689 kPa (6·8 kgf/cm^2) is attained, then allow the oxygen to escape slowly; repeat four times. Bring the oxygen pressure to a value as shown below:

Room Temperature °C	*Pressure*	
	kPa	(kgf/cm^2)
17 to 20	586	5·9
20 to 23	593	5·9
23 to 27	600	6·0
27 to 30	607	6·1
30 to 33	614	6·1
33 to 37	621	6·2
37 to 40	627	6·3

6.2.1 Allow the bomb to stand overnight to make sure that there are no leaks.

NOTE — It has been found that pressure readings as shown above will result in a pressure reading of 758 ± 14 kPa (7·5 ± 0·1 kgf/cm^2) when the bomb is placed in the bath (*see* **6.3**), and consequently no release of oxygen will be required in most cases. This minimizes the chance of a leak developing at the valve after the overnight check for leaks has shown the bomb to be satisfactory.

6.3 Place the bomb in the oil bath maintained at a temperature of 99±0·5°C. As the pressure rises, if needed, intermittently release oxygen from the bomb until a constant pressure 758 ± 14 kPa (7·5 ± 0·1 kgf/cm^2) is obtained and maintained for at least 2 h. A gradual drop in pressure indicates a continuous leak in the bomb. Observe and record the pressure at least every 24 h. In case a leak develops, do not report the results but repeat the test.

6.4 Start timing at the moment of immersion of the bomb in the oil bath and continue the oxidation for the time period specified.

Note — Requirements are usually given in terms of pressure drop in kilopascals (kPa) or kgf/cm^2 at one or more time intervals, for instance, after 100, 200 h, etc.

7. REPORTING

7.1 Report the average of duplicate determinations as pressure drop in kilopascals (kPa) or kgf/cm^2 for the specified test time, or times in hours.

8. PRECISION

8.1 The results of duplicate tests shall not differ by more than the following amounts:

Mean Pressure Drop kPa (kgf/cm^2)	*Repeatability*	*Reproducibility*
0 to 34·5 (0 to 0·34)	13·8 (0·15)	20·7 (0·20)
Over 34·5 to 68·9 (0·34 to 0·68)	20·7 (0·25)	34·5 (0·35)
Over 68·9 to 138 (0·68 to 1·37)	41·4 (0·40)	55·2 (0·60)
Over 138 to 379 (1·37 to 3·77)	68·9 (0·70)	138 (1·40)

Note — These precision values apply only to that portion of the data for which oxygen is absorbed at a rate approximately proportional to time, and before the induction period occurs, as evidenced by a rapid acceleration in the rate of oxygen absorption in a short time interval.

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